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## MEMORANDUM

DATE. 28 December 1998

TO. David Bennett, WAM, U.S. EPA, Region X

FROM: Michelle Turner, Chemist, WESTON, Seattle  
*rum* Roger McGinnis, Senior Environmental Chemist, WESTON, Seattle

SUBJECT: Validation of Organotin Data  
Laboratory Batch: K9805693  
Site: Duwamish River

WORK ASSIGNMENT NO: 46-35-0JZZ

WORK ORDER NO : 4000-019-038-5200-00

DOC. CONTROL NO.: 4000-019-038-AAAK

cc: Bruce Woods, RAP-WAM, U.S. EPA, Region X  
Dena Hughes, Site Manager, WESTON, Seattle (memo only)  
Kevin Mundell-Jackson, Database Management, WESTON

The quality assurance review of four sediment samples, laboratory batch K9805693, collected from the Duwamish River has been completed. The sediment samples were analyzed for organotins by Columbia Analytical Services of Kelso, Washington. Samples were analyzed by gas chromatography with an FPD detector. The samples were numbered.

98344086

98344091

98344095

98344098

### Data Qualifications

The following comments refer to the laboratory performance in meeting the quality control criteria described in the technical specifications of the laboratory subcontract. The review follows the format described in the *National Functional Guidelines for Organic Data Review* (EPA OSWER Directive 9240.1, February 1994), modified to include specific requirements of analytical methods.

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Site. Duwamish River

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1. Timeliness

Holding time limits of 7 days for sample extraction and additional 7 days for analysis were established in the project Sampling and Analysis plan. All samples met holding time criteria

2. Detection Limits

Detection limits met project required quantitation limits.

3. Initial Calibration

A six-point initial calibration was performed prior to each analytical batch. The percent relative standard deviation for the initial calibration was within limits of less than 25 percent RSD

4 Continuing Calibrations

Continuing calibration check was performed after every 10 samples. All target analytes were within required limits for the continuing calibrations with the percent difference for a mid-range standard less than 25 percent.

5. Blanks

a) Laboratory Method Blanks

Laboratory method blank frequency criteria were met. No target analytes were reported in laboratory method blanks.

b) Field Blanks

No field blanks were associated with this SDG

6 Surrogate Compound Recovery

Surrogate recovery goals for Tripropyltin were established in the project Sampling and Analysis Plan at 60 to 130 percent for sediment. Based on conversations with the laboratory an additional surrogate, Triphenyltin was added and historical laboratory

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control chart limits were also used for data qualification. Laboratory limits are presented below:

Surrogate Compound	Sediment Limits
Tripopyltin	20 - 195%
Tripenytltn	20 - 172%

Surrogate compound percent recovery met quality control criteria for all samples, with the exception of the following:

Sample	Surrogate	% Recovery
K9805624-006DMS	Tripenytltn	NA

The Tripenytltn surrogate recovery in the Batch QC was not calculated because of matrix interferences. As surrogate recoveries for all samples in this SDG were within QC limits, no qualifiers were assigned based on surrogate recoveries.

## 7 Laboratory Control Sample (LCS)

LCS recovery goals for Butyltins were established in the project Sampling and Analysis Plan at 60 to 130% for sediment. Based on conversations with the laboratory, historical control chart limits of 20 to 164 percent for sediment were also used for data qualification

Laboratory control sample percent recoveries met QC guidelines (P-project, L-laboratory), with the following exceptions:

LCS	Analyte	% Recovery	QC Limit	Associated Samples
K980827-LCS	Dibutyltin	40	60-130 (P) 20-164 (L)	98344086 98344091 98344095 98344098
K980827-LCS	n-Butyltin	20	60-130 (P) 20-164 (L)	98344086 98344091 98344095 98344098

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Sample results were qualified as estimated (J) when LCS recoveries were outside project limits. Undetected results were qualified as estimated (UJ) when LCS recoveries were outside project limits.

8. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

The following matrix spike recovery goals were established in the project Sampling and Analysis Plan at for sediment.

Analyte	% Recovery
Tributyltin	40 - 120%
Dibutyltin	30 - 120%
n-Butyltin	10 - 120%

Batch MS/MSD sample percent recoveries for Tetrabutyltin met QC guidelines. The relative percent difference (RPD) for Tetrabutyltin was 75 percent. Batch MS/MSD recoveries and RPDs were not calculated for Tributyltin, Dibutyltin and n-Butyltin as the analyte concentration was significantly higher than the spike level. No qualifiers were assigned solely on Batch MS/MSD results.

9. Field Duplicate Analysis

No field duplicates were associated with this SDG.

10. Sample Analysis

A cursory review of raw data was performed. Deliverables were accurate and complete. A duplicate analysis was performed on 98344086. RPDs between replicates were all greater than 35 percent as follows:

Analyte	RPD
Dibutyltin	36
n-butyltin	46

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Results for Dibutyltin and n-Butyltin in sample 98344086 were qualified as estimated. The case narrative indicates that the MS/DMS recoveries of mono-, di- and tributyltin for the Batch QC were not calculated as the analyte concentrations were significantly higher than the added spike solution. The high analyte levels prevented accurate evaluation of the spike recovery. Also, due to this high analyte concentration and matrix interference, surrogate recoveries for the DMS were not calculated. No other problems were noted.

#### 11. Laboratory Contact

No laboratory contact was required.

#### Data Assessment

Upon consideration of the data qualifications noted above, the data are ACCEPTABLE for use except where flagged with data qualifiers that modify the usefulness of the individual values

#### Data Qualifiers

- U - The compound was analyzed for, but was not detected
- UJ - The compound was analyzed for, but was not detected. The associated quantitation limit is an estimate because quality control criteria were not met.
- J - The analyte was positively identified, but the associated numerical value is an estimated quantity because quality control criteria were not met or because concentrations reported are less than the quantitation limit or lowest calibration standard
- R - Quality control indicates that data are unusable (compound may or may not be present). Resampling and reanalysis are necessary for verification
- N - Presumptive evidence of presence of material (tentative identification)
- I - Elevated reporting limit due to matrix interference.

# COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment

**Service Request:** K9805693  
**Date Collected:** 8/20/98  
**Date Received:** 8/21/98

### Butyltins

Sample Name 98344086 Units ug/Kg (ppb)  
 Lab Code K9805693-001 Basis Dry  
 Test Notes D

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	10	10	8/25/98	8/29/98	ND	
Tri-n-butyltin	Method	Butyltins	10	10	8/25/98	8/29/98	42	
Di-n-butyltin	Method	Butyltins	10	10	8/25/98	8/29/98	13	J
n-Butyltin	Method	Butyltins	10	10	8/25/98	8/29/98	10	J

D The MRL is elevated because of matrix interferences and because the sample required diluting

7/12/12/98

Approved By



Date

10/15/98

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# COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment

**Service Request:** K9805693  
**Date Collected:** 8/20/98  
**Date Received:** 8/21/98

### Butyltins

Sample Name 98344091 Units ug/Kg (ppb)  
 Lab Code K9805693-006 Basis Dry  
 Test Notes D

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	10	10	8/25/98	8/29/98	ND	
Tri-n-butyltin	Method	Butyltins	10	10	8/25/98	8/29/98	18	
Di-n-butyltin	Method	Butyltins	10	10	8/25/98	8/29/98	ND	1065
n-Butyltin	Method	Butyltins	10	10	8/25/98	8/29/98	ND	↓

D The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By



Date

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## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment

**Service Request:** K9805693  
**Date Collected:** 8/20/98  
**Date Received:** 8/21/98

## Butyltins

**Sample Name** 98344095 **Units** ug/Kg (ppb)  
**Lab Code** K9805693-010 **Basis** Dry  
**Test Notes** D

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	10	10	8/25/98	8/29/98	ND	
Tri-n-butyltin	Method	Butyltins	10	10	8/25/98	8/29/98	29	
Di-n-butyltin	Method	Butyltins	10	10	8/25/98	8/29/98	ND	10 ug
n-Butyltin	Method	Butyltins	10	10	8/25/98	8/29/98	10	J

D

The MRL is elevated because of matrix interferences and because the sample required diluting

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## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

**Client:** Roy F Weston, Inc  
**Project:** Duwamish River/4000-027-001-2019-38  
**Sample Matrix:** Sediment

**Service Request:** K9805693  
**Date Collected:** 8/20/98  
**Date Received:** 8/21/98

## Butyltins

**Sample Name** 98344098 **Units** ug/Kg (ppb)  
**Lab Code** K9805693-013 **Basis** Dry  
**Test Notes** D

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	10	10	8/25/98	8/29/98	ND	
Tri-n-butyltin	Method	Butyltins	10	10	8/25/98	8/29/98	46	
Di-n-butyltin	Method	Butyltins	10	10	8/25/98	8/29/98	11	J
n-Butyltin	Method	Butyltins	10	10	8/25/98	8/29/98	12	J

D The MRL is elevated because of matrix interferences and because the sample required diluting

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Approved By

*[Signature]*

Date

10/19/98

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